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Initial investigations of lead chromate road paint in the Midwest, United States

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ABSTRACT

Lead chromate ($PbCrO_4$) lacks systematic monitoring data in road paint in the U.S.A. A survey of road paint across Ohio, Indiana, Illinois, Kentucky, and West Virginia was conducted to assess if $PbCrO_4$ exists in road paint in the modern environment. Scanning electron microscopy – energy dispersive spectroscopy (SEM-EDS) indicates Pb-Cr compounds occur in 30 samples among all states. Pb-Cr-bearing particles typically appear as nanometer to micrometer scale particles or aggregates. When present, dissolution textures in sampled road paints are indicative of potential metal release. XRD analysis on five representative paint samples confirms the presence of $PbCrO_4$. Results indicate $PbCrO_4$ is a legacy pollutant in YRP in some regions of the Midwest U.S. $PbCrO_4$ may be transported and systematic geographic studies which integrate materials science research are warranted.

1. Introduction

Heavy metal pollution is a pervasive environmental and human health issue that exists on local, regional, and global scales (Laidlaw et al., 2012; Dietrich et al., 2022; Istanbullu et al., 2023; Panda et al., 2023). This is particularly the case in urban centers where environmental contamination is more likely to occur and where populations continue to grow (White et al., 2014; Lee et al., 2016; Laidlaw et al., 2017; Dietrich et al., 2019; United Nations, 2019; O'Shea et al., 2021a,b). Within this context, two heavy metals of high concern are lead (Pb) and chromium (Cr) as both of these metals pose a significant risk to human and environmental health (Agency for Toxic Substances and Disease Registry (ATSDR), 2012; White et al., 2014; Laidlaw et al., 2017; Meza-Figueroa et al., 2018; O'Shea et al., 2021b; Panda et al., 2023). From the substance priority list as defined by the Agency for Toxic Substances and Disease Registry (Agency for Toxic Substances and Disease Registry (ATSDR), 2022), these ranked at number 2 and number 17 respectively.

Pb has been extensively shown to be a bioaccumulative metal in humans and various other flora and fauna (Lee et al., 2019; Agency for Toxic Substances and Disease Registry (ATSDR), 2020). Long-term Pb exposure in humans may result in a myriad of adverse health effects including negative effects on the gastrointestinal tract, kidneys, skeletal, cardiac, reproductive, and nervous systems (Alatise and Schrauzer, 2010; Laidlaw et al., 2017; Boskabady et al., 2018; Agency for Toxic Substances and Disease Registry (ATSDR), 2020). Pb is a highly problematic environmental contaminant especially for children as they are more vulnerable to Pb ingestion through frequent hand-to-mouth activities. Additionally, the toxic effects of Pb may be exacerbated by children's more vulnerable central

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nervous system and higher adsorption efficiency of their digestive tract as compared to adults (Needleman, 2004; Ko et al., 2007; Stewart et al., 2014; Huang et al., 2016). For children, it is commonly asserted that there are no safe levels of Pb exposure with even relatively low-level Pb exposure in the environment proving to be detrimental (Lanphear et al., 2005; Grandjean, 2010; Grandjean and Landrigan, 2014; Gostin, 2016; Raj and Das, 2023; Zheng et al., 2023).

In the United States (U.S.), common sources of exposure to Pb in the environment have historically included oil, coal, and leaded gasoline combustion. This is in addition to more common, modern-day sources such as Pb pipes, Pb-based paints, historic leaded gasoline, industrial activities (e.g., nonferrous metal mining, smelting, metal manufacturing), Pb-contaminated drinking water, soils, and dust (e.g., Ryan et al., 2004), and other products such as cosmetics (Lanphear et al., 1996; Beak et al., 2008; Laidlaw and Filippelli, 2008; Bocca et al., 2014; Henry et al., 2015; Laidlaw et al., 2017; Boskabady et al., 2018; Dignam et al., 2019; Agency for Toxic Substances and Disease Registry (ATSDR), 2020; Gyamfi et al., 2023; Mostafa et al., 2025). In rural and urban populations, municipal and residential drinking water sources are major pathways through which Pb exposure can occur in the U.S., as Pb is easily transported in water due to its solubility (Triantafyllidou et al., 2009; Santucci and Scully, 2020; Gibson et al., 2024; Tully et al., 2024). For humans, the most common route of Pb exposure is via the digestive and respiratory tracts through ingestion or inhalation. While exposure may also occur via dermal contact, this is less common (Lanphear et al., 1996; Laidlaw et al., 2017; Agency for Toxic Substances and Disease Registry (ATSDR), 2020; O'Shea et al., 2021a; Akhtar et al., 2022; Niemeier et al., 2022).

Much like Pb, Cr is also known to cause adverse health effects in humans (Costa and Klein, 2008; Agency for Toxic Substances and Disease Registry (ATSDR), 2012). While Cr(III) is an essential nutrient in the human body in small amounts, Cr(VI), or hexavalent chromium, is well-recognized as toxic. As a result, it may negatively impact the respiratory tract, the stomach and small intestine, and reproductive systems in humans (e.g, Costa and Klein, 2008; Agency for Toxic Substances and Disease Registry (ATSDR), 2012; White et al., 2014; O'Shea et al., 2021b; Hossini et al., 2022; Mortada et al., 2023; Gautam et al., 2024). Chromium (VI) is a well-documented carcinogen, causing cancers of the lungs, stomach, and intestinal tract with carcinogenic effects on the respiratory system being the most notable in those who have been subjected to long-term aerial particle exposure in occupational settings (Agency for Toxic Substances and Disease Registry (ATSDR), 2012; White et al., 2014; O'Shea et al., 2021b; Alur et al., 2024). Chromium(VI) in the environment is commonly derived from anthropogenic sources such as metal alloys, manufacturing processes, leather tanning, wood preservatives, paints, and anticorrosive agents (Agency for Toxic Substances and Disease Registry (ATSDR), 2012; O'Shea et al., 2021b), as well as from natural sources such as water runoff from ultramafic rocks (Morrison et al., 2009; Manning et al., 2015; Guo et al., 2024). The most common route of Cr exposure in humans is via inhalation in occupational settings (e.g., those employed in metallurgy, tanning industries, or other industries where Cr is utilized in the manufacturing of goods; Agency for Toxic Substances and Disease Registry (ATSDR), 2012; White et al., 2014; O'Shea et al., 2021b; Jiang et al., 2024). Much like Pb, other potential exposure pathways for Cr(VI) include ingestion of contaminated drinking water and soils, as well as dermal contact with contaminated materials (Agency for Toxic Substances and Disease Registry (ATSDR), 2012; Moffat et al., 2018; Georgaki et al., 2023; Ning et al., 2025).

Due to the known detrimental environmental and human health impacts of both Pb and Cr(VI), these two metals are extensively regulated by various U.S. health agencies to reduce environmental and human exposure. However, despite the regulation of Pb and Cr (VI) in the U.S., historic use of these metals in different industrial contexts (e.g. paints, industrial activity) has allowed them to remain persistent in some parts of urban environments (Peña-Fernández et al., 2014; White et al., 2014; Kumar et al., 2017; O'Shea et al., 2021b; Li et al., 2024).

A prime example of this is the historic use of $PbCrO_4$ as a pigment in yellow road paints (YRPs) in the U.S., which has resulted in this Pb-Cr-bearing paint still being observed in the modern environment (White et al., 2014; O'Shea et al., 2021b; Dietrich et al., 2022). In some YRP, $PbCrO_4$ is coated with silica which may reduce the pigment's ability to dissolve or abrade (Pier et al., 1991; Andrady, 1997; Lee et al., 2016; O'Shea et al., 2021b), however, it is unclear when utilizing these coatings first came into practice. Additionally, dissolution or abrasion may still occur despite these coatings when YRP is exposed to different environmental conditions (Triantafyllidou et al., 2009; White et al., 2014; O'Shea et al., 2021b).

Abrasion and/or dissolution of $PbCrO_4$ from YRP presents the opportunity for human exposure through particulate inhalation or ingestion at local and regional scales (Triantafyllidou et al., 2009; White et al., 2014; O'Shea et al., 2021a,b). In addition, abrasion of $PbCrO_4$-bearing road paint via public roadway use and resurfacing is highly probable. $PbCrO_4$ is known to have increased solubility with exposure to 0.05–0.25 M NaCl and $CaCl_2$ road treatments (i.e., road salt and brining treatments; White et al. 2014). This process subsequently creates a mechanism for the introduction of $PbCrO_4$ to the environment, particularly surface and groundwater systems (Triantafyllidou et al., 2009; White et al., 2014; O'Shea et al., 2021a). On a regional scale, transport of eroded or dissolved $PbCrO_4$-bearing road paints into nearby stormwater discharge drains may cause accumulation of $PbCrO_4$ in aquatic environments and may subsequently affect municipal drinking water systems (Triantafyllidou et al., 2009; O'Shea et al., 2021b). Additionally, $PbCrO_4$ in aquatic sediments may persist for a substantial period and may be transported by a wide array of flora and fauna, which can later enable exposure to humans via the consumption of bioaccumulated Pb and Cr(VI) (Lee et al., 2019). From a recent study of European road paints across 11 countries (including blue, green, red, white, and yellow varieties), Turner and Filella (2023) reported lead chromate(s), present at concentrations up to 20 wt%. In addition, Pb was reported in $>$ 50 % of studied paints while Cr was reported in $\sim$ 34 % of studied paints. $PbCrO_4$ was most prevalent in the yellow paint samples.

There is no publicly available registry for the use of $PbCrO_4$ in road paints nationwide throughout the U.S., thus the prevalence and occurrence of $PbCrO_4$-bearing YRP is not quantified. To better understand the current prevalence and potential health hazard presented by $PbCrO_4$ in YRPs in the U.S., this study provides textural, chemical, and mineralogical analysis of suspected $PbCrO_4$-bearing road paints obtained from various municipalities in the Midwestern U.S. (e.g., OH, IN, IL, WV). The primary goals of this study are to 1) determine whether $PbCrO_4$ is present in YRP in communities across the Midwestern U.S., and 2) identify where further assessments of road paints are needed such that the potential environmental and human exposure risk in various localities may be determined.

2. Methods

2.1. Sampling

Road paint samples ($n = 59$) were collected from various locations in Ohio ($n = 15$), Illinois ($n = 19$), Kentucky ($n = 10$), West Virginia ($n = 8$), and Indiana ($n = 7$, Table 1,2; Fig. 1) over multiple days. Sample locations were selected based on safety, occurrence, and availability of potential sample material. The nature of each sample site was variable in both paint condition and in the availability of desired sample mass. Paint fragments were loose or dislodged with clear spatial associations to the road surface materials. Size of paint fragments varied from several millimeters to a few centimeters in maximum diameter and approximately 1 mm to several mm in thickness. Paint fragments were visually contiguous. Sampled materials are large enough for representative comparison by scanning electron microscopy (SEM). Of all samples collected, 56 paint samples were yellow. When present, additional colors were sampled, resulting in the collection of one red, one white, and one green paint sample were also collected, which provided the opportunity to investigate the potential presence of $PbCrO_4$ in other pigments. Samples were collected by carefully collecting paint chips from public road curbs, parking lots, and trafficked roads using clear plastic spoons and gloved hands (Fig. 1). Once a sample was collected, the sample was placed into a clear plastic sample bag and labeled accordingly. GPS coordinates were recorded at each sampling location. To ensure no cross-contamination between sample sites occurred, new gloves and spoons were used at each sampling location.

2.2. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) imaging and energy dispersive spectrometry (EDS) analyses were completed using a Zeiss Supra 35 VP field emission scanning electron microscope (SEM) at the Miami University Center for Advanced Microscopy and Imaging (CAMI). Unmodified road paint samples were first mounted on aluminum SEM stubs with carbon adhesive tabs, and samples were carbon coated. Variable pressure (VP) mode was used with nitrogen (N_2) as the compensating gas to reduce electron charging while imaging sample material. Images were taken using a backscatter detector (BSD) which facilitated the identification of Pb and Cr-bearing particles as heavier elements appeared brighter. $PbCrO_4$ particles are resolvable on this instrument to diameters of 40 nm (e.g, Fig. 5 of White et al., 2014). Dissolution features are assessed by texture. A Bruker Quantax energy dispersive spectrometer (EDS) with a detection limit of approximately 0.1 wt% (Kuisma-Kursula, 2000) was utilized for the acquisition of X-ray spectral analysis for all 59 road paint samples. This sample preparation method and subsequent approach to characterization has been extensively utilized in a variety of similar environmentally focused investigations (e.g., LeGalley and Krekeler, 2013; White et al., 2014; Dietrich et al., 2018; Dietrich et al., 2019), including other pollution-related investigations (Klein and Krekeler, 2020; Flett et al., 2021; O'Shea et al., 2021a; Allen et al., 2024).

2.3. Powder X-ray diffraction (XRD)

Following SEM-EDS analysis, five representative paint samples (MOMG8, YP7, YP20, IOMG5, FILMG3) from localities throughout OH, IN, and IL which were confirmed via SEM to contain Pb and Cr-rich particles, as well as one sample (YP2) which did not contain

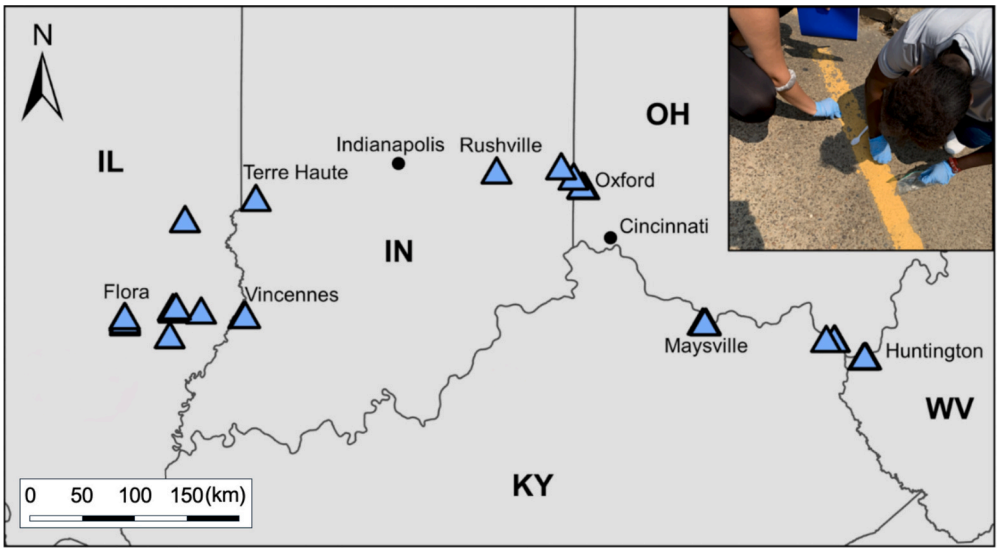

Fig. 1. A location map of all samples taken (blue triangles) with an image demonstrating the road paint sampling method utilized. Owing to scale, some sample location symbols overlap. For exact GPS locations refer to Table 1. As appropriate, personnel wore high visibility vests, gloves, and eyewear with cones and spotters present.

any Pb or Cr (as determined via SEM-EDS) were selected for powder (XRD) using a Bruker D8 Advance X-ray diffractometer at the Department of Geology and Environmental Earth Science at Miami University. Each sample was carefully hand-powdered using an agate mortar and pestle, and samples were analyzed as pack mounts. Samples were scanned from 5° to 65° 2θ with a step size of 0.01° 2θ at a dwell time of 1 s/step. Each sample was analyzed twice for a total run time of 1 h and 43 min per run. The DIFFRAC.EVA Open Crystallography Database in tandem with the RRUFF Project Database Powder Diffraction data were utilized to identify major to minor mineral peaks for each sample analyzed (Lafuente et al., 2015; Table 1).

3. Results

SEM-EDS analysis indicates that the chemical compositions of yellow, white, red, and green road paint vary (Figs. 2–4). This elemental variation between road paint sample types indicates that they are composed of mixtures of several different minerals and chemical compounds (Figs. 2–5).

Red road paint as observed by SEM-EDS is chemically dominated by Si, O, Al, Ca, and Fe with minor Zn peaks present (Fig. 2A). White road paint tends to be compositionally dominated by Si, Mg, Na, Al, and Ca with major to minor peaks of Ti and Fe (Fig. 2B). Green road paint is compositionally dominated by Si, Mg, and Ca with major to minor peaks of Ti, Fe, and Cu. Pb and Cr-bearing particles also appear in the green road paint sampled as aggregates dispersed throughout the paint matrix (Fig. 2C, 3C).

Texturally, red, green, and white road paint samples vary. The red road paint sample contains anhedral quartz (SiO_2), anhedral to subhedral rutile (TiO_2), and euhedral to subhedral calcite ($CaCO_3$) crystals dispersed throughout the paint matrix. Glassy spherules on the scale of < 10 μm to > 100 μm are also present throughout the sample with Ca and Si being the dominant chemical components of these spherules (Fig. 2A, 3A). White road paint also contains glassy spherules varying in size throughout the paint matrix with the majority of spherules exceeding 100 μm in diameter (Fig. 2B, 3B). While some spherules display minor pitting (Fig. 3B), most are smooth with no indication of significant abrasion or erosion (Fig. 2B, 3B). Other materials identified within the paint matrix of white road paint include anhedral quartz and euhedral to subhedral calcite as well as amorphous Fe- and Ti-oxides (Fig. 2B). In green road paints, common textures as indicated by SEM analysis include glassy spherules ranging in size from approximately 2 μm to 50 μm in diameter. Anhedral quartz and subhedral to euhedral calcite are also present. Green road paints have minor amounts of Cu, however a specific pigment phase is not identifiable. SEM-EDS analysis indicates that green road paint also contains minor amounts of Pb and Cr interspersed throughout the paint matrix, and in these cases, the Pb- and Cr-rich particles appear as 10 to 20 μm aggregates of elongate, anhedral crystals that are usually less than 1 μm in diameter (Fig. 3C). $PbCrO_4$ cannot be reliably identified as the primary mineral phase for these Pb- and Cr-bearing particles in green paint, as sample mass is limited and thus unsuitable for XRD analysis.

The overall chemical composition of YRP is dominated by Si, Ca, O, Al, and Mg with peaks of Ti and Fe varying in intensity throughout the sample suite. Out of all YRP analyzed via SEM-EDS, Pb, and Cr were present in $n = 30$ samples, including 7 samples

Table 1
Sample ID, location, paint color, and presence of Pb and Cr as detected (or not) via SEM-EDS.

Sample ID	City	State	Color	$PbCrO_4$	Sample ID	City	State	Color	$PbCrO_4$
YP1	Oxford	OH	Yellow	No	IOMG2	Ironton	OH	Yellow	Yes
YP2	Oxford	OH	Yellow	No	IOMG3	Ironton	OH	Yellow	No
YP3	Oxford	OH	Yellow	No	IOMG4	Ironton	OH	Yellow	Yes
YP4	Oxford	OH	Yellow	No	IOMG5	Ironton	OH	Yellow	No
YP5	Oxford	OH	Yellow	Yes	IOMG6	Ironton	OH	Yellow	Yes
YP6	College Corner	IN	Yellow	No	IOMG7	Ironton	OH	Yellow	No
YP7	Liberty	IN	Yellow	Yes	IOMG8	Ironton	OH	Yellow	Yes
WP8	Liberty	IN	White	No	IOMG9	Ironton	OH	Yellow	No
YP9	Olney	IL	Yellow	No	IOMG10	Ironton	OH	Yellow	Yes
YP10	Olney	IL	Yellow	No	MOMG1	Maysville	KY	Yellow	No
YP11	Flora	IL	Yellow	Yes	MOMG2	Maysville	KY	Yellow	No
YP12	Vincennes	IN	Yellow	No	MOMG3	Maysville	KY	Yellow	Yes
YP13	Vincennes	IN	Yellow	Yes	MOMG4	Maysville	KY	Yellow	Yes
YP14	Sumner	IL	Yellow	No	MOMG5	Maysville	KY	Yellow	Yes
YP15	Olney	IL	Yellow	No	MOMG6	Maysville	KY	Yellow	Yes
RP16	Olney	IL	Red	No	MOMG7	Maysville	KY	Yellow	Yes
YP17	Olney	IL	Yellow	No	MOMG8	Maysville	KY	Yellow	Yes
YP18	Casey	IL	Yellow	No	MOMG9	Maysville	KY	Yellow	Yes
YP19	Terre Haute	IN	Yellow	Yes	MOMG10	Maysville	KY	Yellow	No
YP20	Rushville	IN	Yellow	Yes	FILMG1	Flora	IL	Yellow	Yes
WVMG1	Huntington	WV	Yellow	Yes	FILMG2	Flora	IL	Yellow	Yes
WVMG2	Huntington	WV	Yellow	Yes	FILMG3	Flora	IL	Yellow	Yes
WVMG5	Huntington	WV	Yellow	No	FILMG4	Flora	IL	Yellow	Yes
WVMG6	Huntington	WV	Yellow	No	FILMG5	Flora	IL	Yellow	Yes
WVMG7	Huntington	WV	Yellow	Yes	FILMG6	Flora	IL	Yellow	No
WVMG8a	Huntington	WV	Yellow	Yes	OIMG1	Olney	IL	Yellow	No
WVMG8b	Huntington	WV	Green	Yes	OIMG2	Olney	IL	Yellow	No
WVMG9	Huntington	WV	Yellow	No	OIMG3	Olney	IL	Yellow	No
WVMG10	Huntington	WV	Yellow	No	OIMG4	Olney	IL	Yellow	No
IOMG1	Ironton	OH	Yellow	Yes	OIMG5	Olney	IL	Yellow	Yes

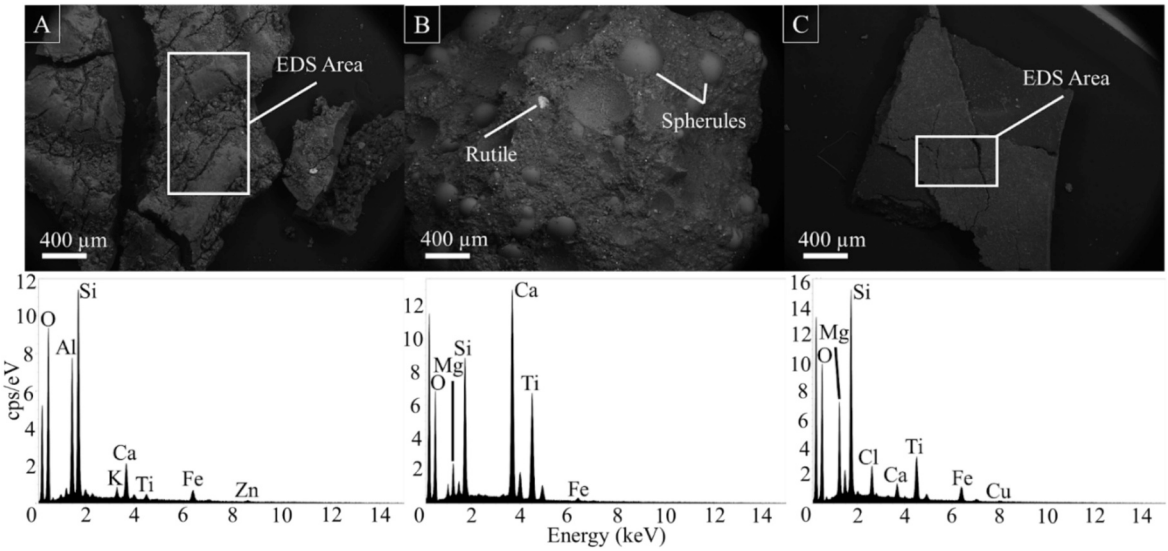

Fig. 2. Low magnification SEM images of red, white, and green road paints (top) with respective EDS spectra (bottom). A) Red road paint taken from Olney, IL. EDS indicates that red road paint is primarily composed of Si, O, Al, and Ca with major to trace amounts of Ti, Fe, and Zn present. B) White road paint taken from Liberty, IN. Sample displays large spherules interspersed throughout the matrix. EDS indicates that the sample is composed primarily of Ca, Si, Mg, Ti, and O with major to trace amounts of Fe present. C) Green road paint taken from Huntington, WV. EDS indicates major amounts of Si, Mg, O, Ti, and Cl with trace Ca, Fe, and Cu.

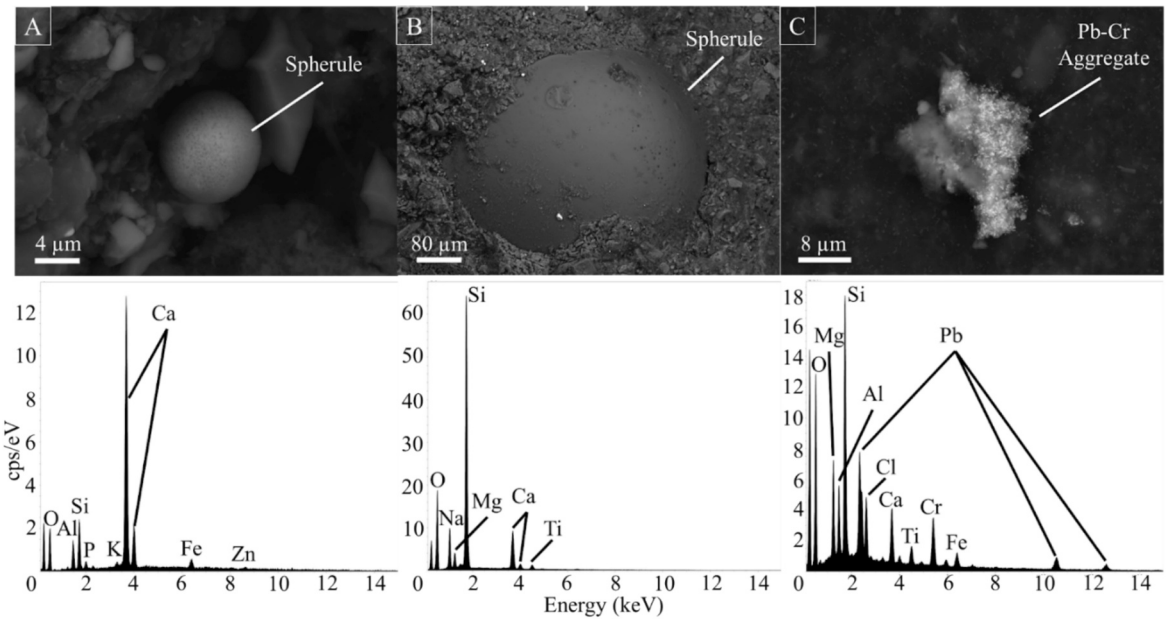

Fig. 3. High magnification SEM BSD images of red, white, and green road paints (top) with respective EDS spectra (bottom). A) Spherule identified in red road paint taken from Olney, IL. EDS indicates that this spherule road paint is primarily composed of Ca, Si, Al, and O with major to trace amounts of Fe, and Zn present. B) Spherule identified in white road paint taken from Liberty, IN. This spherule is approximately 480 µm in diameter and is primarily composed of Si and O with major to minor peaks of Ca, Na, Mg, and Ti present. C) $PbCrO_4$ aggregate identified within green road paint taken from Huntington, WV. EDS indicates major amounts of Si, O, Mg, Al, Cl, Ca, Cr, Ti, and Fe.

from IL, 4 samples from IN, 14 samples from OH, and 4 samples from WV (Tables 1–2).

Texturally, YRPs are typically composed of subhedral to euhedral quartz and calcite crystals suspended within an organic paint matrix, along with amorphous Fe- and Ti-oxide materials (Fig. 4, 5). Glassy spherules also occur throughout the paint matrix in several samples. They typically vary in size from ~ 1 µm to > 100 µm in diameter and are primarily composed of Si and O (Fig. 4A, 5A). Significant textural variation between Pb-Cr particles within YRP paint samples is also observed (Figs. 4–5). Pb-Cr particles occur in

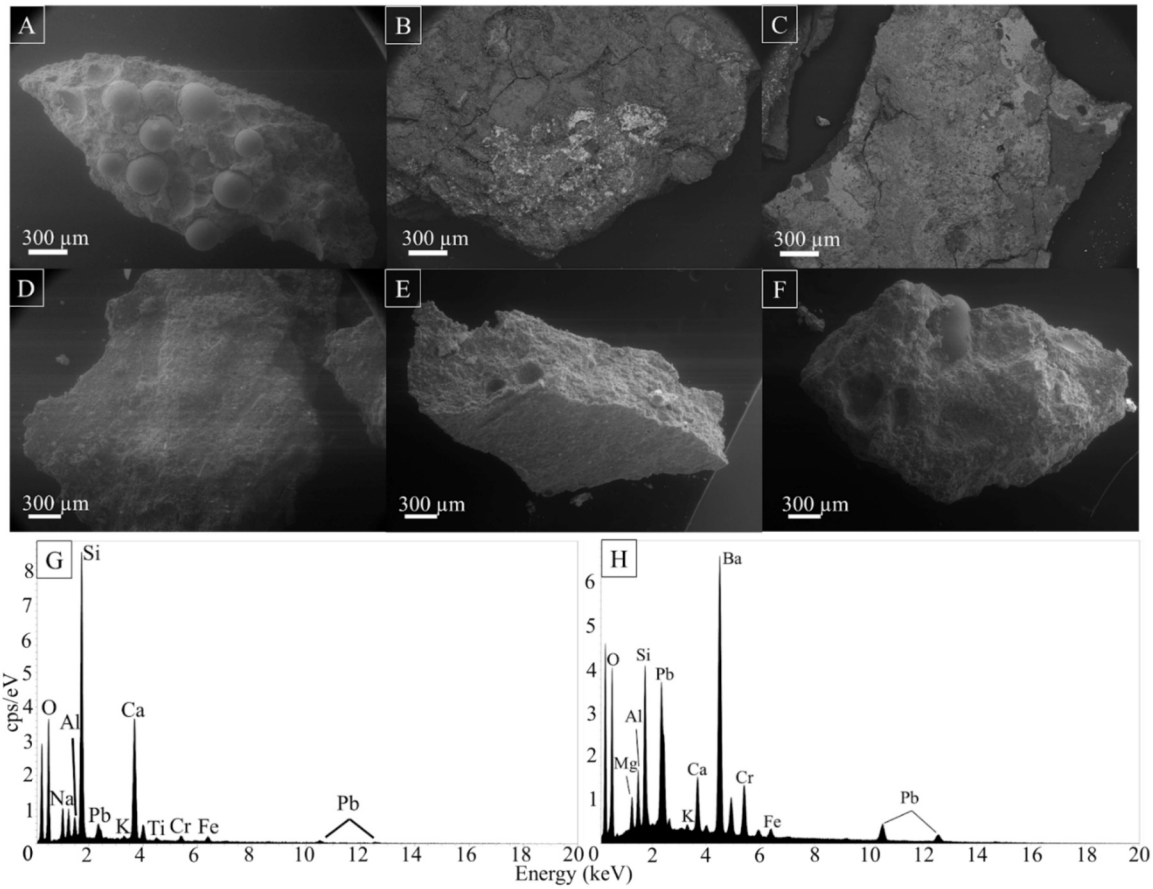

Fig. 4. Low magnification SEM-BSD images of yellow paint chips with representative EDS displaying common elements found in most samples as well as those containing $PbCrO_4$. A) Nearly botryoidal paint chip sample containing $PbCrO_4$ from Terre Haute, IN. B) Multilayer paint chip from Flora, IL displaying $PbCrO_4$ (bright region). Note, $PbCrO_4$ is not present in all layers. C) Multilayer paint chip containing PbCrO4 taken from Liberty, IN. D) Multilayer paint chip containing $PbCrO_4$ taken from Rushville, IN. E) Paint chip containing $PbCrO_4$ taken from Huntington, WV. F) Paint chip containing $PbCrO_4$ taken from Huntington, WV. G) Representative EDS spectra of sample A. EDS shows trace Pb and Cr, with Si, Ca, and O being other common elements present within the sample. H) Representative EDS spectra of C. EDS shows Ti, Si, O, Pb, and Cr are prevalent in this sample.

these samples most often as either subhedral or euhedral crystal aggregates that vary in diameter from < 10 µm to > 20 µm with single crystals approximately 0.1 to 0.2 µm in width and 0.2 to 0.4 µm in length being less common (Fig. 5B, C).

Powder XRD allows for the identification of major mineral phases in selected YRP samples (e.g., MOMG8, YP7, YP20, FILMG3, IOMG5, and YP2). Powder XRD allows for the identification of the mineral crocoite ($PbCrO_4$) as the common mineral phase associated with YRP samples as determined by SEM-EDS. In addition, XRD analysis was utilized to determine the mineralogical composition of inorganic materials dispersed throughout the paint matrix. Sample YP2 does not contain any Pb-Cr-bearing particles as observed by SEM-EDS and was selected for powder XRD analysis for general comparison of the inorganic constituents of YRP.

Basic powder XRD patterns show that calcite is the primary inorganic component in YRP with major reflections of (012), (104), (110), (113), (202), (018), and (116) in all samples analyzed. Quartz, dolomite, and rutile are also present in all samples analyzed, however peak intensity varies. With respect to quartz, all samples have major to minor reflections of (100) and (101), and samples YP20 and FILMG3 have an additional reflection of (112). In all samples analyzed, rutile shows major to minor reflections of (110) and (211), and though additional reflections of (101) and (111) may occur, there is some overlap of peaks for calcite (110) and dolomite (113), respectively. Dolomite is identifiable in all samples by a reflection of (104) (Fig. 6).

Of the five Pb-Cr-bearing YRP samples analyzed via XRD, crocoite (or $PbCrO_4$), is observed as a major constituent of YP7, YP20, and FILMG3 with major reflections of (200) and (120). Crocoite peaks of ($\underline{2}$ 02) and ($\underline{1}$ 32) are also observed in FILMG3, however, these peaks can not be reliably identified in YP7 or YP20 (Fig. 6). Though Pb-Cr-bearing particles are observed in MOMG8 and IOMG5 via SEM-EDS, crocoite peaks can not be reliably identified via powder XRD.

4. Discussion

Pb-based paint pigments are a well-recognized pervasive problem in urban environments (e.g., Jacobs 1995; Jacobs et al., 2002;

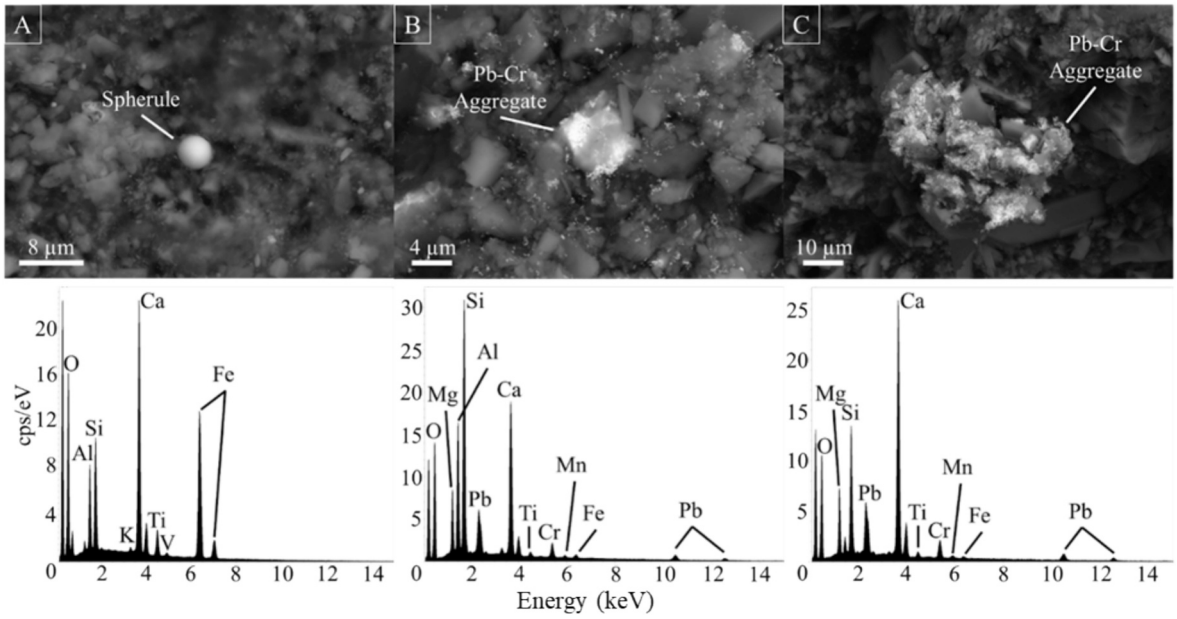

Fig. 5. High magnification SEM BSD images of yellow paint chips from Maysville, OH with respective EDS spectra. A) Glassy spherule approximately 4 µm in diameter primarily composed of Fe and O surrounded by Si, Ca, Al rich matrix material. Minor amounts of Ti and V are also present. B) Pb-Cr aggregate containing small amounts of Mn, Fe, and Ti surrounded by Si, Mg, Al, and O rich paint matrix. C) Pb-Cr aggregate surrounded by Ca, Si, O, and Mg rich matrix. Minor Ti, Mn, and Fe are also present within the matrix.

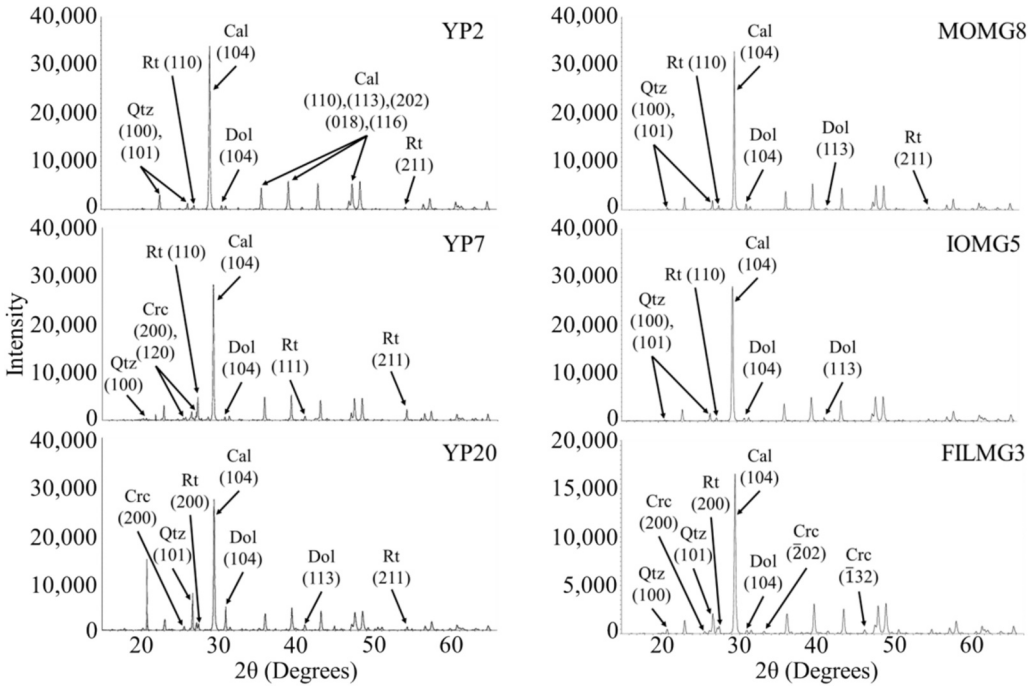

Fig. 6. Basic powder XRD patterns for yellow paint samples YP2, YP7, YP20, MOMG8, IOMG5, and FILMG3. Major peaks for calcite (Cal), quartz (Qtz), dolomite (Dol), rutile (Rt), and crocoite (Crc), or $PbCrO_4$, are labeled with corresponding Miller indices.

O'Connor et al., 2018; Ranjbar et al., 2023). In 1978, the United States banned the use of Pb-based paint in residential homes, and as such Pb-based house paint remains a well recognized and on-going issue (e.g., Schwartz and Levin, 1991; Jacobs et al., 2002; O'Connor et al., 2018; Clickner et al., 2020, US EPA, 2024). Specific exposure problems with Pb-based paint are detailed in older literature, for example: Spittler and Feder (1979) examined Pb uptake in urban gardens traced to widespread existence of Pb-based paint in Boston,

Massachusetts. Clark et al., 2006 later investigated exposure pathways in urban gardens in Massachusetts and found that Pb-based paint contributed 40 to 80 % of the mass balance in vegetation. Clark et al. (2008) expanded on that research with a larger study on urban gardens in Roxbury and Dorchester, Massachusetts and reported that Pb derived from paint was a major pollutant. Chaney et al. (1984) conducted a similar study in Baltimore, Maryland and, in addition to Pb-based paint being a source of Pb in urban areas, pollution from traffic was also a concern. In 1996, the final steps were taken to finalize the ban on the use and sale of leaded gasoline and was considered "one of the great environmental achievements of all time" (US EPA, 1996).

While no evidence to support the occurrence of lung cancer in workers exposed to $PbCrO_4$ has been found, the National Institutes of Health (NIH) report it to be "toxic by ingestion and inhalation" with potential carcinogenesis induced by $PbCrO_4$ as a result of centrosome dysfunction (National Industrial Chemicals Notification and Assessment Scheme (NICNAS), 2015). Within this context, the bioaccessibility of $PbCrO_4$ is therefore important to consider with the work of Rinner (2011) suggesting that multiple factors play a role in the cause of its toxicity, including particulate purity, size, shape, and surface area. While early reports by the IARC on chromium compounds concluded they be considered human carcinogens, mutagenicity tests produced variable results with the bioavailability of these compounds an open question (International Agency for Research on Cancer (IARC), 1980, 1987). Later, Connor and Pier (1990) reported that by encapsulating the pigments in amorphous silica, compounds became "non-mutagenic and non-toxic" thereby reducing the bioavailability of the active toxic species. While more recent work directly addressing the toxicity and bioavailability of $PbCrO_4$ is limited, the work of Samaradiwakara et al. (2025, and works cited within) reported the potential for Pb to enter local soils through the weathering of Pb-bearing house paint and the production of Pb-bearing nanoparticles. Documenting the presence, and characterizing the nature, of Pb-bearing compounds in the environment is therefore warranted.

This investigation of road paint across the Midwest U.S. is therefore important in examining a commonly overlooked source of Pb. It demonstrates the complexity of the inorganic mineralogical components of these materials, and documents the variability amongst color-based types (e.g., white, green, red, yellow paints). In terms of inorganic paint components, all of the paints sampled contain mineral or glass phases (e.g., calcium silicate glass, calcite, quartz, dolomite) that are generally considered to be environmentally benign. Despite this, some paints ($n = 30$; Table 1) contain Pb- and Cr-bearing particles, although the mechanisms by which these compounds may break down in the natural environment is not the focus of this study.

The common inorganic phases of calcite, quartz, and Fe-and Ti-oxides identified in YRP, although not of major environmental concern, may contribute to road sediment contamination through mechanical and chemical weathering of these paints. A recent US road sediment review by Dietrich et al. (2022) highlighted the need for better constraints on the nature of road sediment contaminant sources such that tracing of pollution sources could be improved. Whether the calcite present in road paint is a natural mineral or a chemical precipitate cannot be determined in this study, thus the application of isotopic tracer studies utilizing Pb, Cu, Zn, and Sr would likely be an appropriate next step congruent with road sediment studies as proposed in Dietrich et al. (2022) and Gokey et al. (in review). Pb isotopic analyses of road sediment in this study's sample region, as described in a similar study by LeGalley et al. (2013); e.g., $^{208}Pb/^{204}Pb$, $^{206}Pb/^{204}Pb$, may also help to determine the contribution of $PbCrO_4$ from YRP as an environmental pollutant. The characterization of road paints presented here therefore provides the initial textural, mineralogical, and chemical context for subsequent bulk isotopic analyses in support of future investigations of road paints in other areas of the U.S. outside of the Midwest. Collectively, this work would further our understanding of YRP provenance and the nature of metals and elements present in all road paints in the modern environment.

Most glassy spherules are interpreted to be glass microbeads which are common additives to road paints (Burghardt et al., 2022; Turner and Filella, 2023). For example, glass spherules are added to improve reflectance and visibility at night (Stoudt and Vedam, 1978). Determining the source(s) of the glass spherules, however, cannot be determined with SEM-EDS analysis alone as the sources of glassy spherules in the natural and built environment may vary (Kutchko and Kim, 2006; Shetye et al., 2019; Allen et al., 2024). Although it is reasonable to interpret the glassy spherules as intentional additives, another interpretation is that at least some of the spherules may be derived from anthropogenic activity such as coal fly ash, abrasion of vehicle parts, and metal manufacturing processes (e.g., Magiera et al., 2011; LeGalley and Krekeler 2013; Shetye et al., 2019; Allen et al., 2024). This alternative interpretation is supported by the uniform calcium silicate compositions observed via SEM-EDS (Fig. 2B, 3A-B, 4A). Results of this study indicate that detailed investigation of these spherules within road paint and associated road sediment which have well constrained provenance is warranted.

$PbCrO_4$ is identified as the primary mineral phase of Pb- and Cr-bearing particles in 30 paint samples in the Midwest (OH, IL, IN, and WV) by the occurrence of Pb and Cr in SEM-EDS imaging and spectra, and observation of major $PbCrO_4$ peaks in three samples as observed in XRD data (Fig. 6). Additionally, $PbCrO_4$ particles as observed in SEM are texturally similar to those previously reported in other studies where $PbCrO_4$ was observed in YRP (White et al., 2014; O'Shea et al., 2021a). In addition to 28 YRP samples, one green paint sample was found to contain Pb- and Cr-bearing particles, however the identification of $PbCrO_4$ as the primary mineral phase is not possible in this study due to the limited amount of sample material obtained. Historically a variety of Pb- and Cr- inorganic compounds have been utilized in green pigments in paints such as $PbCrO_4{\cdot}PbSO_4 + FeNH_4Fe(CN)_6$, to create chrome green pigment (Gettens and Stout, 1966; Sward, 1972). Despite records indicating that these compounds have been commonly used as paint pigments, the extent of use of these pigments in green (and other) road paint is unclear.

The spatial variance of $PbCrO_4$-bearing road paint across all sampling locations is attributed to various degrees of repainting over old $PbCrO_4$-bearing YRP with $PbCrO_4$-free paint, or painting new road surfaces with $PbCrO_4$-free paint. Examples of mixed $PbCrO_4$-bearing YRP with $PbCrO_4$-free paint are common within collected samples (Fig. 2C; Fig. 4C). This paint mixing has the potential to create additional complexity within the context of tracing road sediment pollution sources (e.g., via Pb isotopes, see above), as well as through bulk chemical analysis using methods such as XRF or ICP-MS. Identifying the presence of $PbCrO_4$ as a discreet mineral phase in road paints is best approached through detailed, high-resolution SEM-EDS imaging techniques as this permits the detection of low

amounts of Pb and Cr (~0.1 wt%; Kuisma-Kursula, 2000). The identification of $PbCrO_4$ as the primary Pb-Cr-bearing compound must then be confirmed using basic powder XRD techniques (White et al., 2014). It should be noted, however, that issues may arise if the mixed nature of YRP is not acknowledged as there is a risk where a relatively small contribution from YRP may not fully be captured in a sample despite the presence of $PbCrO_4$-bearing paint within a larger region. Due to this compound layering effect in YRP, the proportions of $PbCrO_4$-free and $PbCrO_4$-bearing road paint are likely highly variable across a given region. Additionally, this study has shown that Pb- and Cr-bearing green road paint exists (Fig. 2C). However, it is generally unknown if this paint is pigmented with $PbCrO_4$ alone, or if other Pb-Cr-bearing compounds such as those in chrome green pigment have been utilized (e.g., $PbCrO_4{\cdot}PbSO_4 + FeNH_4Fe(CN)_6$; Turner and Solman, 2016; Turner and Filella, 2023). While SEM-EDS analysis confirmed that the green road paint analyzed in this study did contain Fe, no S peaks were identified, therefore the presence of chrome green as the primary inorganic pigment in the paint sampled in this study is unclear.

Due to the compound layering of paint, as well as the potential age range of $PbCrO_4$-bearing YRP, future investigations via transmission electron microscopy (TEM) is recommended. This would enable corrosion textures, dissolution-reprecipitation textures, and minerals (or phases) present at lower abundances and/or sizes to be detected. Identification of such features would allow constraints to be placed on the processes associated with YRP breakdown, resulting in a better understanding of the contributions of $PbCrO_4$-bearing YRP to the environment. For example, White et al. (2014) documented dissolution or alteration textures in $PbCrO_4$ in YRP from Hamilton, OH, and attributed this (at least in part) to winter road treatments using NaCl and $CaCl_2$ compounds. At present, the extent, nature of dissolution or corrosion, and whether there is reprecipitation of $PbCrO_4$ (or other Pb- or Cr-bearing phases) remains largely unconstrained. O'Shea et al. (2021b) recently investigated $PbCrO_4$-bearing YRP in simulated environmental and biological fluids, and evaluated the potential for the release of Pb and Cr via inductively coupled plasma-optical emission spectrometry (ICP-OES). Their study indicated that little (ingestion) to no (environmental and inhalation) Pb and Cr leaching occurred across three different experimental set-ups. This lack of elemental leaching was attributed to the presence of silica coatings which are utilized in $PbCrO_4$-bearing YRP to minimize mechanical and chemical weathering (i.e., the formation of Pb- and/or Cr- rich fragments produced via abrasion, chemically-aided dissolution, and physical wear; White et al., 2014, Turner and Filella, 2023). From an earlier study by LeGalley and Krekeler (2013), Pb in the urban environment from northern Hamilton, OH was proposed to be predominantly associated with the dispersal of $PbCrO_4$-bearing YRPs. When combined with the work of White et al. (2014), it is therefore likely that road treatments do play a role in weathering of YRP and subsequent dispersal of $PbCrO_4$ in the environment. However, mechanical weathering and dispersal of fine micrometer to sub-micrometer $PbCrO_4$ material by runoff and wind action is also highly likely to affect the fate and transport of Pb and Cr in the environment.

Overall, the potential contribution of Pb and Cr from $PbCrO_4$-bearing YRP, the variable extent of $PbCrO_4$ weathering, the reprecipitation of $PbCrO_4$, and/or the (re)crystallization of other Pb- or Cr- phases, may vary based on climate patterns and environmental conditions on local and regional scales. This potential for $PbCrO_4$ in all road paints to weather in the natural environment may therefore pose a risk to those who may be exposed to particles in these settings. In these cases, the size of these $PbCrO_4$ particles must also be taken into account. Particulate matter (PM) < 10 μm are considered to be respirable and have the potential to affect deep lung tissue while PM < 2.5 μm has the potential to enter the bloodstream (US EPA, 2024). $PbCrO_4$ particles observed via SEM-EDS in this study typically occur in aggregates which vary from $<$ 10 to 20 μm in diameter with individual Pb-Cr-bearing particles often being 0.1–0.2 μm in width and 0.2–0.4 μm in length (Fig. 3C, 5B-C). While O'Shea et al. (2021b) found that limited Pb or Cr leaching occurred in simulated lung fluids, the size of the particles observed in this study indicate that they may still be respirable. Thus, detailed investigations of $PbCrO_4$-bearing YRP throughout the U.S. is warranted using the same techniques as LeGalley and Krekeler (2013) and White et al. (2014), in combination with TEM in order to better understand dissolution, corrosion, and reprecipitation processes of $PbCrO_4$ and related phases. It is also recommended that investigations similar to that of O'Shea et al. (2021b) be undertaken using $PbCrO_4$-bearing YRP from a wide range of environments (e.g., urban, rural, humid, desert) to determine the potential for these paints to degrade in varying environmental conditions and evaluate the potential human health effects.

This investigation of the Midwestern U.S. samples provides a robust framework for future studies in the U.S. It is noted that Turner and Filella (2023) recently completed a similar detailed exploratory study of Pb and Cr in European road paints and documented the common occurrence of $PbCrO_4$ as a pigment. Turner and Filella (2023) also note variation in the inorganic compounds utilized as pigments in these paints (e.g., chrome green – $PbCrO_4{\cdot}PbSO_4 + FeNH_4Fe(CN)_6$, white lead – $2PbCO_3{\cdot}Pb(OH)_2$) and thus strengthen the need for more detailed studies of YRP and other road colored paints using a combination of electron microscopy methods and Pb-isotopic source apportionment mixing models.

Urban hydrology is a well-recognized complex system with pollution being a major issue for water resources (e.g., Fletcher et al., 2013). Well established options exist for capturing roadway stormwater runoff (e.g., Maestri and Lord, 1987; Wong et al., 2000), including catchment basin filtration (Alam et al., 2018). Specifically the use of sand filtration alone (e.g., Zarezadeh et al., 2018) and sand and mixed sand biochar filters (Koivusalo et al., 2023) have shown improved filtration results (over 80 % Pb removal), and could be implemented to mitigate runoff of dissolved and particulate $PbCrO_4$ road paint particles. Furthermore, other approaches such as porous concrete containing iron slag with a sand filter adsorbent (Koupai et al., 2016) may be useful to mitigate surface and groundwater pollution. Ebihara et al. (2009) investigated treatment of contaminated roadway runoff using vegetated filter strips and found net particle retention was found to be greater than 70 % for particles of 0.020 mm or larger. Considering such technological approaches for remediation, the fine particle size observed for $PbCrO_4$ particles in road sediment should be accounted for along with the solubility of $PbCrO_4$ in road treatment solutions (e.g., White et al., 2014). This investigation of $PbCrO_4$ particles found in road paint, provides context for future studies attempting detailed experiments investigating break through curves for both particulate transport and solute transport for granular media approaches such as sand and mixed sand biochar filters, and may provide some context for vegetation based approaches as well.

As Pb and Cr^{6+} are well-recognized to be of concern within the context of environmental health, this investigation provides a mineralogical and chemical perspective on the nature and occurrence of these potential pollutants in urban and rural transportation systems. Many of the locations selected by this study are relatively small urban population centers surrounded by rural settings. Whether or not there is more $PbCrO_4$-bearing YRP in either of these settings is unclear but could be addressed by more extensive sampling surveys (i.e., higher density sampling). This study therefore provides a comparative framework for future surveys of $PbCrO_4$-bearing YRP in the U.S. and other countries.

5. Conclusions and implications

Lead chromate ($PbCrO_4$) is detected and remains persistent in ~ 50 % of road paint samples ($n = 30$) collected in this study as indicated by SEM EDS and XRD analysis. $PbCrO_4$ is therefore likely present in a range of rural and urban settings throughout the Midwest, U.S., due to the historic use of $PbCrO_4$-based YRP paint. The presence of $PbCrO_4$ paint, and their PM size, ultimately poses an environmental exposure risk via atmospheric interactions, road sediment build-up, and stormwater runoff in regions where these paints are present.

It is recommended by this study that future road paint studies integrate transmission electron microscopy (TEM) with SEM EDS and XRD to allow for high magnification imaging in combination with the acquisition of diffraction patterns. These could then be used to determine the crystallinity of minerals throughout the paint matrix, identify the nature of potential Pb-Cr-phases, and evaluate dissolution reactions. Such constraints would enable a better understanding of processes in YRP evolution and environmental dispersal. Combined with a high sampling density, bulk sample analyses via X-ray fluorescence (XRF) and/or high resolution inductively coupled plasma – mass spectrometry (ICP-MS) would permit quantification of elements from the wt. % to the ppt level within a highly spatially-constrained context. While this approach would quantify the absolute concentrations of certain heavy metals within bulk road paint samples, in samples where $PbCrO_4$-pigmented paint is mixed or repainted with $PbCrO_4$-free paint, bulk chemical analysis may not be a reliable method by which to determine the presence of $PbCrO_4$ and its availability to the environment.

While significant efforts to characterize road paints are still needed at local, regional, and global scales, the existence and persistence of bioaccumulative metals like Pb and Cr in YRPs in the Midwest, U.S. presents a topic of environmental concern. The full extent of the impact of $PbCrO_4$ across a range of natural and built environments (e.g., desert, humid) and communities (e.g., rural, urban) is currently poorly constrained, thus, highly spatially constrained studies which integrate the mineralogical, elemental, and isotopic approaches proposed here are recommended.

CRediT authorship contribution statement

Kailee J. Gokey: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Claire L. McLeod:** Writing – review & editing, Visualization, Supervision, Methodology, Investigation. **Kinshuk Tella:** Writing – original draft, Investigation. **Morgan Gillis:** Writing – original draft, Methodology, Investigation. **Mireille Fouh Mbindi:** Methodology, Investigation. **Marion L. Lytle:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **Mark P.S. Krekeler:** .

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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